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# DEMONSTRATION OF A PROTOTYPE HYDROGEN SENSOR AND ELECTRONICS PACKAGE--Progress Report 2

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## DEMONSTRATION OF A PROTOTYPE HYDROGEN SENSOR AND ELECTRONICS PACKAGE

### Progress Report

SCAQMD Contract No. 16039

Reporting time period: March 27, 2016 to May 26, 2016

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SCAQMD Project Officer: Lisa Mirisola

SCAQMD Contract Administrator: Drue Hargis

Task	Completion by
1. Station Demonstration and Site Recommendation	3/26/16
2. Order Sensor Equipment	3/26/16
3. Build Sensors	5/26/16
4. Install Sensors	7/26/16
5. Sensor Demonstration and Data Analysis	1/26/17

Table 1. List of contract tasks and expected completion dates

### Project Establishment

The subcontracting process between LLNL and LANL was completed on February 23, 2016.

#### Task 1: Chino was selected for the second site demonstration.

During our visit on April 27<sup>th</sup>, we received permission to install a sensor at the Hyundai Hydrogen Station in Chino from Dan Poppe and are planning for an installation date in early July.

We met with David Blekhman, Technical Director, and Michael Dray, Technical Operations & Hydrogen Station Manager at the Cal State L.A. Hydrogen Research and Fueling Facility (H2 Station) on Thursday, April 28<sup>th</sup>. During this visit, we discussed potential sensor locations and decided upon a configuration with the sensor located on top of the dispenser with an option to install tubing to measure hydrogen concentrations in the dispenser well as needed (Fig. 1). Due to the additional funding need to secure collaboration with David Blekhman and his group, we have decided to retain this option as a back up.





Fig. 1. Dispenser and exposed well (left) and LLNL/LANL team with David Blekhman at the Cal State L.A. Hydrogen Research and Fueling Facility

### **Task 2: All sensor and platform materials were ordered.**

As of March 27<sup>th</sup>, LANL researchers placed an order with Custom Sensor Solutions for three constant resistance sensor power supplies and impedance buffer units, acquired chromite, metal oxide type H<sub>2</sub> sensor elements from ElectroScience and ordered e-beam shadow masks to fabricate the LLNL/LANL H<sub>2</sub> sensor elements based on ITO for Task 3.

### **Task 3: Three sensor units were prepared.**

The field trials unit to be deployed at Chino has been deposited and assembled and is presently being tested at LANL. The Chino unit currently employs an ESL-fabricated, lanthanum chromite sensor element for testing but may be updated with a LANL-fabricated, ITO-based sensor element of the type tested at Burbank during the DOE-funded field trials work conducted in 2014-2015. Parts for an additional fuel trials unit are presently being machined and assembled, and additional ITO sensors are being prepared in order to optimize the fabrication process.

Further optimization of these units is being actively explored. LANL researchers, Eric Brosha and Chris Romero, are working with Dr. William Buttner at NREL Hydrogen Sensor Testing Laboratory to test a complete sensor package using standard DOE hydrogen sensor testing protocol at no cost. In May, LANL sent NREL a lanthanum chromite type sensor, fabricated by ESL and recovered from Burbank in April. This sensor ran continuously from August 2015 to April 2016 at H2F.

LANL requested a modification to the standard test procedure to include additional experiments that will aid in decoupling cyclic weather events from thermal shifts to determine whether the oscillating baseline is caused by a lack of thermal compensation in the electronics. NREL will also perform tests in which the sensor baseline stability is measured during programmed changes in relative humidity, pressure and temperature independently. These results will provide guidance for future sensor selection and improvements to be incorporated in next generation electronics.

The lanthanum chromite type electrode was developed in response to previous NREL testing to address survivability during the most harsh testing conditions performed during testing with DOE standard protocols: H<sub>2</sub> exposure during anaerobic conditions. This experiment will be performed again to confirm that the lanthanum chromite type electrochemical H<sub>2</sub> safety sensor offers a solution for operational conditions that may require periods of anaerobic operation caused by oxygen starvation or depletion during a catastrophic station event.

LANL designed and built an adapter to permit a field trials unit to sample from a small, remote enclosure, such as the lower cabinet in the Cal State dispenser, and has measured effect of flow rate through the modified field trials unit. Flow impedance increased, reducing the turnover rate inside the enclosure by almost 1/3. The 3/8" OD tygon tubing will be replaced with 1/2" OD and the adapter will be modified accordingly. During these experiments, fine particles of activated carbon used to adsorb contaminants in the field were observed; these could dislodge from the filter area and block flow of sample gas to the sensor. LANL is presently improving the contaminant trap for the follow-on deployment at the Chino station.

LANL fabricated an enclosure to simulate sampling from the bottom portion of the Cal State dispenser where H<sub>2</sub> is released during vehicle refueling. Various leaks were simulated over a weekend where H<sub>2</sub> release duration was varied while the signal was recorded from the field trials sensor using the wireless system. The remote sampling adapter for Cal State was tested in this simulation.

In the meantime, LLNL is preparing for indium tin oxide (ITO) and yttria-stabilized zirconia (YSZ) depositions via pulsed power magnetron sputtering. Five YSZ core tape with thin coatings of MgAl<sub>2</sub>O<sub>4</sub> spinel with Pt pads and heater were sent by LANL for deposition trials (Fig. 2). While comparing the performance of chromite vs. indium tin oxide electrodes is outside the scope of this study, it is of significant interest in sensor optimization and this work can inform future sensor field trials studies.

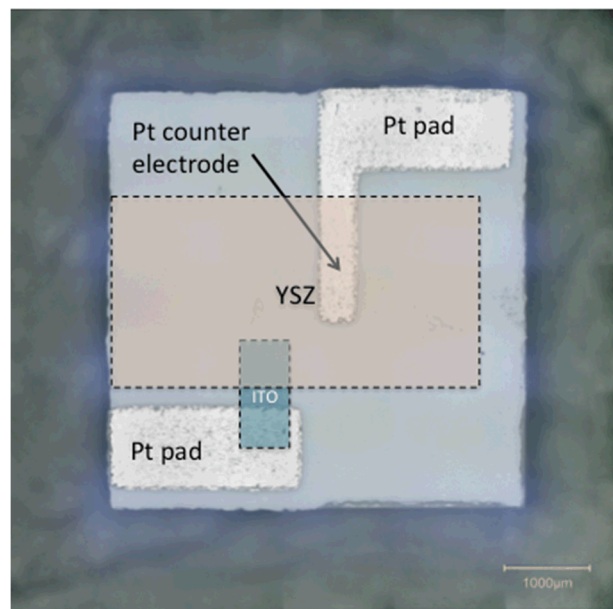


Fig. 2. Optical micrograph of sensor platform with marked regions for ITO and YSZ deposition.

LANL is also pursuing ITO deposition. Three ITO working electrodes were deposited on next generation ESL sensor platforms and one device was completed with a YSZ electrolyte layer. The device exhibits unusually high impedance and the YSZ deposition mask geometry was modified to increase electrolyte coverage of the electrodes. New shadow masks are being designed and fabricated to change the electrode spacing and to better define electrolyte geometry. A second complete ITO-YSZ sensor is being prepared.

#### **Task 4: Maintenance was performed on sensors at Burbank.**

We replaced the two sensor elements, wireless transmitter batteries, weather station console battery, charcoal filters and a broken ethernet cord at the City of Burbank Hydrogen Refueling Station. Both sensors are currently operational and logging data.

#### **Task 5: Data logging at Burbank was initiated on April 27<sup>th</sup>.**

We have achieved continuous operation of two field trials units installed at Burbank in April. As of June 10, the sensor has performed 45 days of continuous operation with no false alarms or wireless signal dropouts. The station has been offline through May, so no fill events have been recorded. However some trace releases of H<sub>2</sub> were recorded and no sensor baseline drift has occurred at either the compressor or compressor skid location.

In data collected from April 27<sup>th</sup> through May 23<sup>rd</sup>, weather station data confirmed that the sensor baseline oscillation is commensurate with normal temperature cycles. The experiments performed at NREL should confirm whether the behavior is entirely linked to temperature fluctuations or some other periodic influence such as barometric pressure or relative humidity (all are linked). This behavior was previously observed during the DOE-funded field trial and is attributed to a response of the system electronics, rather than the sensor.

LabView source code developed under the DOE-funded H<sub>2</sub> Safety Sensor field trials work was obtained from the original LabView developer and sent to LLNL to investigate the possibility of making improvements to the control software.

#### **Schedule**

All work is proceeding according to the planned schedule.

	3/26/16	5/26/16	7/26/16	1/26/16
<b>Task 1</b>	□			
<b>Task 2</b>	□			
<b>Task 3</b>		□		
<b>Task 4</b>			X	
<b>Task 5</b>				X

Table 3. Planned (black) and executed (blue) work

#### **Budget**

- \$100,000 subcontract allotment to LANL for sensor materials, deposition and construction, station selection, installation support and monitoring
  - \$9,151.80 was incurred for researcher time, materials and travel to LA for sensor maintenance.
- \$75,000 allotment to LLNL for station selection, sensor monitoring, project management and reporting

- \$934.56 was incurred for travel to LA for sensor maintenance
- \$7,178.76 was incurred for researcher time

Current LLNL personnel expenses and LANL materials expenses and researcher time meet are anticipated to increase during sensor development and monitoring over the next two months.

**Planned Work for May 27, 2016 to July 27, 2016**

- Upgrade LabView software to allow for independent baseline calibration of multiple sensors at a single location (LLNL).
- Deposit indium tin oxide and yttria-stabilized zirconia and evaluate microstructure, thermal stability and robustness (LLNL).
- Complete NREL validation and verification of lanthanum chromite electrode sensors (LANL).
- Install new field trials unit at Chino (LANL-LLNL).
- Complete design and testing of the adapter to permit remote sampling of a confined space.

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